JDB

Site: Syntex Verona
ID #: MOD007452154
Break: 12.8
Other: Spring River
Report 3/17/87

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 7
25 FUNSTON ROAD
KANSAS CITY, KANSAS 66115

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MEMORANDUM

SUBJECT: Data Review Comments for Spring River Dioxin Analysis

FROM: William Bunn for W. Bunn
Acting Chief, CLQA Section

TO: Robert Morby
Chief, SPFD/WSTM

THRU: Robert D. Kleopfer, Ph.D. RD Brown for
Acting Chief, LABO/ENSV

We have completed our review of the dioxin data submitted as part of the annual report of fish and sediment samples for Spring River. Attached are our comments. If you have any questions concerning the review, please let me know.

Attachment

cc: Dale Bates

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MEMORANDUM

TO: R. D. Kleopfer, Acting Chief, LABO

FROM: T. S. Viswanathan

THRU: Jim Jackson, E&E/TATL
Cliff Kirchmer, ATATL

DATE: March 6, 1987

RE: Review of Spring River 2378-TCDD Data
From Syntex Research
TDD# 07-8702-054
PAN# TMO 0061 TAQ

The annual report of analyses of fish and sediment samples taken from the Spring River in 1986 by Syntex Research has been reviewed. The following are our findings:

1. The analytical method to be followed by Syntex Research has been reviewed earlier by me and by Dr. Cliff Kirchmer. The method was scientifically sound and it incorporated quality assurance features comparable to those used by EPA in their contract laboratory program.

2. The company reports data for sediment and fish samples provided by the Missouri Department of Conservation. The fish were collected from 5 sites. One group of samples (Group A) were filleted and only the fillets were analyzed for 2378-TCDD. A second group (Group B) was analyzed for the skinless fillet part and the remainder (the entire fish minus the right fillet). A third group (Group C) utilized the whole fish for the 2378-TCDD analysis. The sample numbers and the amounts of the sixteen fish samples are shown in Table I. The fish homogenates were prepared by the Environmental Trace Substances Research Center in Columbia and sent to Syntex through the University of Nebraska (Lincoln) for 2378-TCDD analysis.

3. The analytical results for 2378-TCDD in the sediment (sample Nos. 1-3) and fish (sample no. 4-19) samples from the Spring River are shown in Table II. The sediments are free of 2378-TCDD. All the fish samples except #017 contain low ppt levels of 2378-TCDD. The sediment samples, however, have high detection limits (2-10 ppt) since they were analyzed using LRMS>

4. The amount of 2378-TCDD in the whole fish was calculated as a "weighted mean" from the known weight of the fillet and remainder fractions and their 2378-TCDD contents. These calculations were done by Syntex scientists and a summary of these results are shown in Table III.

5. Overall, the data appears to be good. The following comments are concerned with the quality assurance procedures followed by the laboratories:

   a. The sediment samples appear to have been analyzed with a LRMS method. The analysts used a 4-point calibration. The samples did not contain any TCDD and the detection limits were calculated using a " 2.5 times noise" method. The responses for native TCDD, when present, were below the "2.5 times noise multiple" criteria. One of the samples (AKJC4-003) was spiked with TCDD prior to analysis and another (AKJC4-001) was analyzed in duplicate. The results shown in Table IV indicate satisfactory results for the quality assurance procedures.

   b. The fish samples have been analyzed using a HRMS method. The analysts used a 5-point calibration. The calibration was excellent with a correlation coefficient close to unity and a standard error estimate in the low third decimal place. One calibration (chromatograms not provided) was run in conjunction with the glassware blanks and another (11/10/86) was run in conjunction with the sample analysis. All the fish extracts were analyzed on 11/10/86 and 11/11/86 with periodic response factor verification steps several times during the day. Two samples were analyzed in duplicate and one sample was spiked with native TCDD and analyzed. These results, which are quite satisfactory, are also shown in Table IV.

   c. The laboratory ran three glassware blanks while they were getting ready for this analyses. The results for these blanks (Table IV) are quite satisfactory. However, there is no indication of any method blanks that were run with the samples. The fish samples were extracted in several batches from 10/20/86 to 11/4/86. Sample sizes of 40 to 50 grams were used with $^{13}C_{12}$-2378-TCDD spikes of 2 ng. The company does not indicate whether they ran one or more laboratory method blanks that went through all the steps that samples normally are subjected to, during the complete analytical procedure. The glassware blanks address only some parts of the overall procedure. Besides, they were run in a time frame (9/19 to 9/25) different from the time frame (10/20 to 11/11) used in the actual sample analyses. The inclusion of such blank data in the report will enhance the quality of the analytical results, which appear to be good in the eyes of this reviewer.

TABLE I

DESCRIPTION OF SPRING RIVER FISH SAMPLES

Site	Group	Sample No. AKJC4-# and Weight					
		Fillets		Remainder		Whole Fish	
		#	Wt.(gms)	#	Wt.(gms)	#	Wt.(gms)
1	B	005	159	006	772	020	931
2	B	009	290	010	1847	021	2137
3	B	012	175	013	1006	022	1181
4	B	015	104	016	687	023	791
5	B	018	260	019	1439	024	1699
1	A	004		---		---	948
1	C	---		---		007	1165
2	A	008		---		---	1689
3	A	011		---		---	1252
4	A	014		---		---	711
5	A	017		---		---	1651

TABLE II

Amount Of 2378-TCDD in Spring River Sediment and Fish Samples

Sample No.	Amount (ppt) ppt	D.L.
AKJC4-001**	ND	(7.5)
-001D**	ND	(6.1)
-002**	ND	(2.6)
-003**	ND	(9.1)
-004	2.8	
-005	2.5	
-006	9.6[t]	
-006D	9.8[t]	
-007	13.2	
-008	2.3	
-009	4.4	
-010	18.9	
-011	1.2	
-012	1.3	
-013	7.1	
-014	1.1	
-015	1.7	
-016	7.7	
-017	ND	(0.66)
-018	1.2	
-019	1.8[t]	
-019D	2.0[t]	
-020	*	
-021	*	
-022	*	
-023	*	
-024	*	

** - Sediment samples
* - Calculated values
t - Mean is used for duplicate runs

TABLE III

2378-TCDD Levels in Spring River Fish Samples

Site	Group	Sample # Whole Fish	2378-TCDD Present (ppt) Fillet	Remainder	Whole Fish*
1	B	AKJC4-020	2.5	9.7	8.5
2	B	-021	4.4	18.9	16.9
3	B	-022	1.3	7.1	6.2
4	B	-023	1.7	7.7	6.9
5	B	-024	1.2	1.9	1.8
1	C	-007	---	---	13.2
1	A	-004	2.8	---	----
2	A	-008	2.3	---	----
3	A	-011	1.2	---	----
4	A	-014	1.1	---	----
5	A	-017	ND(0.66)	---	----

* The amount shown for the whole fish are calculated values except that for sample #AKJC4-007. The following equation was used:

$$\text{Amount} = [(W_f * A_f) + (W_r * A_r)]/(W_f + W_r).$$

W_f = Wt. of fillet; W_r = Wt. of remainder
A_f = Amount of TCDD in fillet; A_r = amount of TCDD in remainder

TABLE IV

Quality Control Measures Included With the Samples

Sample	Amount of 2378-TCDD (ppt)	(D.L.)	Comments
AKJC4 - 001	ND	(7.5)	
- 001D	ND	(6.1)	LRMS method
AKJC4 - 006	9.6		
- 006D	9.8		Mean 9.7; HRMS method
AKJC4 - 019	1.8		
- 019D	2.0		Mean 1.9; HRMS method
Glassware Blanks			HRMS
Columns #3	ND	(0.22)	Run on 9/25/86
Flasks #4	ND	(0.15)	9/25/86
Flasks #5	ND	(0.28)	9/25/86
AKJC4 - 014	1.1		
- 014S	3.7		Sample spiked with 3.48 ppt Native 2378-TCDD;HRMS
AKJC4 - 003	ND	(9.1)	
- 003S	29.5		Sample spiked with 31 ppt Native 2378-TCDD;HRMS
Method Blanks			None Reported
Field Blanks			None Reported